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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF HARD SOLDERS FOR JOINTING ALUMINIUM AND ALUMINIUM ALLOYS

PART II DETERMINATION OF ALUMINIUM

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF HARD SOLDERS FOR JOINTING ALUMINIUM AND ALUMINIUM ALLOYS

PART II DETERMINATION OF ALUMINIUM

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 19 February 1982, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Chemical analysis of hard solders for jointing aluminium and aluminium alloys, the chemical composition of which is specified in IS : 5479-1969*, is covered in two parts of this standard. In this part, method for determination of aluminium is covered. Method for the determination of all other constituents in hard solders for jointing aluminium and aluminium alloys is covered in IS : 8812 (Part I)-1978†.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960‡.

1. SCOPE

1.1 This standard (Part II) covers the method of determination of aluminium in hard solders, the chemical composition of which is given in IS : 5479-1969* (aluminium containing in the range from 4·75 to 31 percent).

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*Specification for solders for jointing aluminium and aluminium alloys.

†Methods of chemical analysis of hard solders for jointing aluminium and aluminium alloys: Part I Determination of silver, copper, zinc, antimony, arsenic, iron and bismuth.

‡Rules for rounding off numerical values (*revised*).

2. SAMPLING

2.1 Laboratory Sample — It shall be drawn and prepared in accordance with IS : 1817-1961*.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF ALUMINIUM BY EDTA METHOD

4.1 Outline of the Method — After dissolution of the sample in the hydrochloric acid, it is fumed with dilute sulphuric acid. Copper, arsenic, antimony, bismuth, iron and zinc are removed by electrolysis in mercury cathode cell. Residual zinc and aluminium are complexed quantitatively with EDTA by boiling with an excess of the reagent. The excess EDTA is back titrated with standard zinc solution at about *p*H 5·2 using xylenol orange as indicator. The aluminium EDTA complex on boiling with ammonium fluoride liberates EDTA quantitatively which when titrated with standard zinc solution gives the amount of aluminium present in the solution.

4.2 Apparatus — Mercury Cathode cell.

4.3 Reagents

4.3.1 *Dilute Hydrochloric Acid* — 1 : 1 and 1 : 9 (*v/v*).

4.3.2 *Concentrated Nitric Acid* — rd = 1·42 (Conforming to IS : 264-1976‡).

4.3.3 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

4.3.4 *EDTA Solution (0·05 M)* — Dissolve about 18·64 g of the disodium salt of EDTA $[(OOC{\cdot}CH_2)_2\ NH{\cdot}CH_2CH_2{\cdot}NH(CH_2COO)_2\ Na_2{\cdot}2H_2O]$ in water and transfer to a 1 000-ml volumetric flask, dilute to the mark and mix.

4.3.5 *Methyl Orange Indicator Solution* — 0·05 percent in water.

4.3.6 *Dilute Ammonia* — 1 : 2 (*v/v*).

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*Methods of sampling non-ferrous metals for chemical analysis.

†Specification for water for general laboratory use (*second revision*).

‡Specification for nitric acid (*second revision*).

4.3.7 *Acetate Buffer Solution* (p*H 5·2*) — Dissolve 21·5 g of sodium acetate (CH_3 COO Na· $3H_2O$) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 litre.

4.3.8 *Standard Zinc Solution* (*1 ml = 3·0 mg of Zn*) — Transfer 3·000 0 g of zinc (purity 99·9 percent *Min*) to a 400-ml beaker, add 150 ml of dilute hydrochloric acid (1 : 4), cover it with a watch-glass and warm gently until the zinc is dissolved. Cool, transfer to a 1-litre volumetric flask, dilute to the mark and mix.

4.3.9 *Xylenol Orange* — Dissolve 0·1g of xylenol orange in 100 ml of water containing two drops of dilute hydrochloric acid (1 : 1).

4.3.10 *Ammonium Fluoride* (*20 percent*) — Dissolve 20 g of ammonium fluoride in 100 ml of water. Add 3 drops of xylenol orange (**4.3.9**) and then dilute hydrochloric acid (1 : 9) dropwise till the colour changes to yellow.

4.3.11 *Standard Aluminium Solution* (*1·0 mg Al/ml*) — Weigh 1·000 g of aluminium metal (99·9 percent purity) into a beaker containing 100 ml of dilute hydrochloric acid (1 : 1), add a small drop of mercury and heat gently until dissolution is complete. Cool, transfer to a 1-litre volumetric flask, dilute to the mark and mix.

NOTE — Mercury will not dissolve and may be removed while transferring the solution to 1-litre flask.

4.4 Procedure

4.4.1 Transfer 1·000 g of the sample to a 400-ml beaker and dissolve in 20 ml of dilute hydrochloric acid (1 : 1). When reaction subsides, add 1 ml of concentrated nitric acid, boil off nitrous fumes and cool. Add 5 ml of dilute sulphuric acid and evaporate just to white fumes. Allow the residue to cool, add 10 to 20 ml of water , rinse down the sides of the beaker with water, and again evaporate to white fumes to remove the last traces of nitric acid. Allow the residue to cool, dilute to 100 ml with water and heat until the sulphates have been dissolved.

4.4.2 Electrolyze the solution overnight in mercury cathode cell at a current of 1 to 1·5 A or electrolyze at a current of 3 to 5 A with vigorous stirring of the mercury and electrolyte, for 3 to 4 hours. Separate the mercury from the electrolyte while at least part of the current is kept on. Filter the electrolyte into a 400-ml beaker and discard the residue.

4.4.3 Dilute the filtrate up to the mark in a 250-ml volumetric flask and mix. Transfer a suitable aliquot containing 20 to 30 mg of aluminium to a 500-ml conical flask, add sufficient EDTA solution to provide an excess of at least 5 ml over the expected amount of aluminium and residual zinc. Add 2 to 3 drops of methyl orange indicator solution and neutralise with dropwise addition of dilute ammonia till the indicator colour changes from red to yellow. Add 25 ml of acetate buffer, and heat to boiling for 5 minutes. Cool, add 10 ml of acetate buffer, wash the sides with distilled water and titrate with standard zinc solution using 5-6 drops of xylenol orange as indicator. The end point is indicated by a clear change from yellow to orange colour. Add 25 ml of ammonium fluoride solution, heat to boiling for 5 minutes. Cool, wash the sides with distilled water, and titrate the liberated EDTA with standard zinc solution to deep orange colour. If titanium is present, necessary correction for titanium in final calculation shall be made.

4.5 Calculation

$$\text{Aluminium, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of standard zinc solution required for the titration of liberated EDTA,

B = equivalent aluminium in g per ml of standard zinc solution, and

C = mass in g of the sample represented by the aliquot of solution taken.